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(54) Method and apparatus for hydrogen peroxide vapor sterilization

(57) In order to sterilize a chamber, gas is circulated through the chamber (1), and through a dehumidifier connected to the chamber. The humidity of the gas is monitored and, when the humidity is sufficiently low, hydrogen peroxide is introduced into the circulating gas until a suitable circulation of hydrogen peroxide in the gas has been reached. That level or greater of hydrogen peroxide is maintained for a suitable time, possibly adding additional hydrogen peroxide to the gas to maintain the level. After that time, the hydrogen peroxide is removed from the gas, for example by passing it over a metal catalyst (103) which separates the hydrogen peroxide into water and oxygen. Preferably, in order to measure the level of hydrogen peroxide in the gas, the gas containing the hydrogen peroxide may be diluted by a known ratio before passing through a sensor (102).

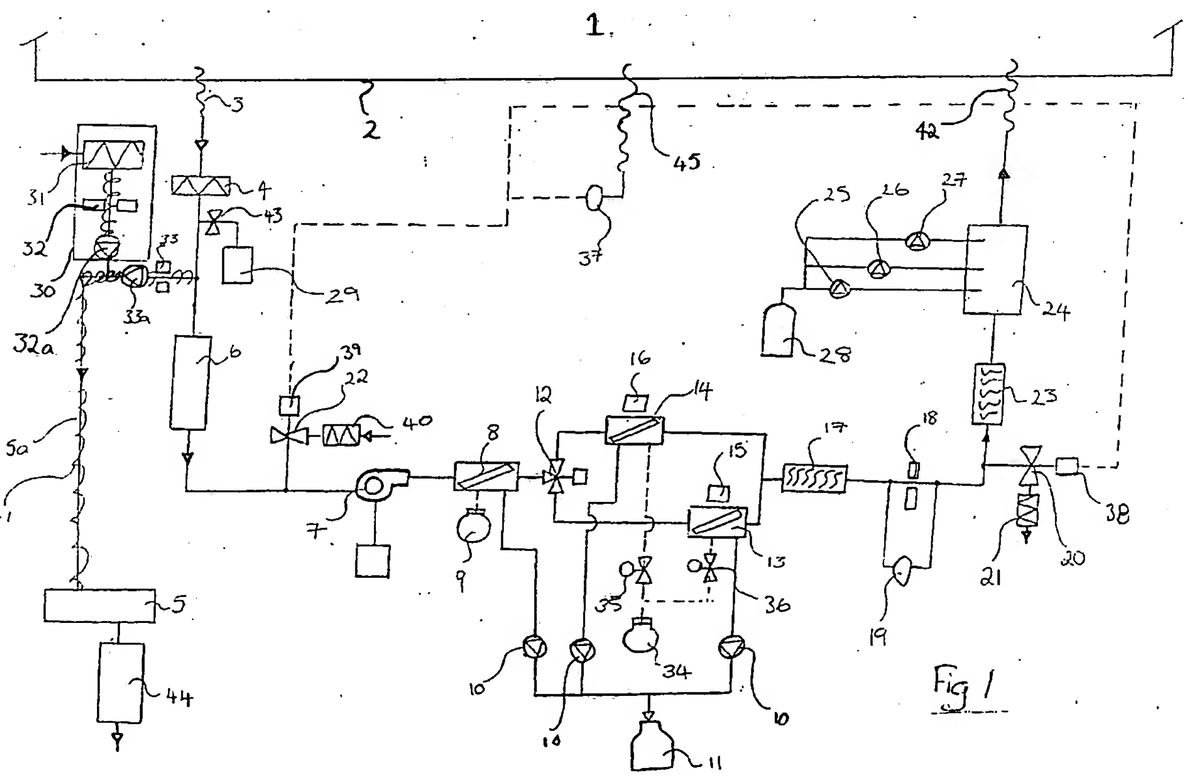

Fig 1

EP 0 774 263 A1

Description

The present invention relates to a method and apparatus for sterilizing a chamber using hydrogen peroxide gas as the sterilant.

The applications for sterile work areas are widespread in the pharmaceutical, biotechnology, and food industries, as well as the medical world. A large number of compounds have been put forward as sterilising agents but most achieve only a sanitization effect and may have serious side effects such as toxicity, corrosion, environmental damage or a combination of these. Formaldehyde has long been used as a cheap and quite effective sterilising agent but doubts over its safety and environmental persistence may prevent continued use.

Hydrogen peroxide is a simple and cheap compound with good sterilising properties. Its major advantage is that it can be decomposed to water and oxygen which are totally harmless products.

In the vapor phase, hydrogen peroxide can be used to treat work areas of size from safety cabinets to cleanrooms. Like all gas phase sterilization, deep layers of contamination will not be affected, but as a surface sterilant, results for hydrogen peroxide look very attractive.

WO 89/06140 (American Sterilizer Company) discloses a sterilization method including the steps of injecting a hydrogen peroxide and water vapor mixture into a sterilization chamber in an initial amount less than the saturation limit of the vapor mixture in the chamber and then injecting a plurality of intermittent make-up injections of the vapor mixture into the chamber in order to maintain the concentration of hydrogen peroxide vapor at a level effective for sterilization but less than that concentration of hydrogen peroxide vapor which would raise concentration of the vapor mixture to the saturation limit. This purports to overcome the problems of hydrogen peroxide vapor degradation and condensation by employing knowledge concerning the changing saturation limit of hydrogen peroxide vapor during the sterilization cycle.

US Re.33,007 (Re-issue of US 4,642,165, American Sterilizer Company) discloses a method of vaporizing a multi-component liquid, for example a binary composition of hydrogen peroxide and water, by injection into a vaporization chamber in open communication with a vacuum chamber. It further discloses monitoring the multi-component liquid from the liquid reservoir to the vaporization chamber by means of a 3-way ball valve. In this way the liquid can be delivered in discrete increments at a predetermined rate onto a heated surface of the vaporization chamber where it is instantaneously vaporized.

US 4,169,123 and 4,169,124 (More et al and Forstrum et al, respectively) describe sterilization techniques using gaseous hydrogen peroxide. A hydrogen peroxide and water solution is vaporized in a closed sterilization chamber. The vapors are permitted to contact the items to be sterilized to achieve sterilization.

The present applicants have produced generators, sold under the trade mark Microflow, which are designed to produce hydrogen peroxide vapor as a gas-phase surface sterilant for volumes from under 1 cubic metre up to cleanrooms of 200 cubic metres. The gas is produced continuously by evaporation of a hydrogen peroxide solution in water, to give peroxide concentrations in the gas up to 4000 ppm (volume/volume) in a closed loop system including the chamber being sterilized, requiring no duct to atmosphere.

It is important that water vapor is removed from the sterilizing chamber as this allows the maximum loading of hydrogen peroxide vapor into the chamber without condensation of water occurring.

In the applicants' generators, water vapor is removed from the air in the system by passing it through a refrigerant dryer and then reheating it. Hydrogen peroxide is then evaporated into the warm dry airstream and the resulting gas mixture is passed into the chamber.

The gas returns to the generator from the chamber by passing through a filter and then through a catalyst which removes all the remaining peroxide, converting it to water vapor and oxygen, recycling clean air to a blower which drives the system.

Throughout this process various assumptions have to be made, for example, the time to achieve a complete air change in the chamber being sterilized, the number of air changes needed to reduce the relative air humidity to the required level, the number of air changes required to increase the hydrogen peroxide gas concentration to the desired level and the number of air changes required to reduce the gas concentration after the sterilization step has occurred.

The number of air changes required to reduce the gas concentration after sterilization is important as hydrogen peroxide is toxic.

The present applicants have found that the calculations relied on to make the various assumptions discussed above are often inaccurate. For example, there are many factors which will influence the time required to remove the hydrogen peroxide gas from the chamber. These factors include gassing of hydrogen peroxide gas from the surfaces of the chamber. Also, some materials, such as flexible PVC, absorb greater quantities of the gas and hence will increase the time required to reduce the gas concentration to an acceptable level.

Also it has now been found that assumptions as to the amount of hydrogen peroxide entering the chamber being sterilized, and hence the hydrogen peroxide concentration in the chamber, are not justified. Losses of hydrogen peroxide occur in the process of generating the gas from a solution in water, in absorption in pipework and other surfaces and by its spontaneous decomposition.

In order to optimize the operation of the system from the viewpoint of both cost and time the present applicants have realised that it is desirable to remove as

many of these assumptions as possible.

According to the invention in one aspect there is provided a method of sterilizing a chamber using hydrogen peroxide as sterilant, in which the gas of the atmosphere in the chamber is cycled through apparatus adapted to dehumidify it, add hydrogen peroxide gas to it and remove hydrogen peroxide gas from it, the method comprising the steps of

(1) dehumidifying the gas in said apparatus and sensing the humidity of the atmosphere in the chamber to detect when a predetermined low humidity value is obtained,
(2) when said predetermined humidity value is obtained, adding hydrogen peroxide gas to the gas in said apparatus and sensing the hydrogen peroxide concentration in the atmosphere in the chamber to detect when a predetermined first hydrogen peroxide concentration value is obtained,
(3) when said predetermined first hydrogen peroxide concentration value has been obtained, maintaining the hydrogen peroxide concentration in the atmosphere -in the chamber at at least a further predetermined value, which may be the same as said first value, for a predetermined period of time, by sensing the hydrogen peroxide concentration in the atmosphere in the chamber and adding further hydrogen peroxide gas to the gas in said apparatus as required, and
(4) after said predetermined period of time, removing hydrogen peroxide from the gas in said apparatus and sensing the hydrogen peroxide concentration in the chamber to detect when a predetermined low value thereof is obtained.

According to the present invention in a second aspect there is provided a sterilizing apparatus for continuously circulating gas, e.g. air, through a chamber to be sterilized, said apparatus comprising

means for circulating the gas;
at least one dehumidifier for the circulating gas;
means for introducing hydrogen peroxide into the gas flow prior to it entering the chamber;
means for removing hydrogen peroxide from the gas flow after it leaves the chamber;
a first sensor for monitoring the relative humidity of the gas in the chamber;
a second sensor for monitoring the concentration of hydrogen peroxide of the gas in the chamber; and
control means adapted and arranged to control said means for introducing hydrogen peroxide in dependence on the outputs of said first and second sensors.

The invention can be employed for various gases in the chamber, but the discussion below will refer to the case when it is air.

The first sensor for monitoring the relative humidity of the air flow is preferably positioned such that air returning to the apparatus from the chamber can be monitored. In this way an accurate measure of the relative humidity of the air in the chamber can be obtained.

In a preferred form, only a sample of the air returning from the chamber is directed to the first sensor to be monitored, while the remainder continues on its cycle thereby causing no interruption to the continuous flow. It is only necessary to monitor the relative humidity of the air prior to the introduction of the hydrogen peroxide and therefore a valve may be provided to control when a sample of air is directed to the first sensor.

It is preferred that the relative humidity of the air in the chamber is less than 20%, ideally less than 10%, prior to the introduction of the hydrogen peroxide.

The second sensor for monitoring the hydrogen peroxide concentration is preferably positioned to monitor the air flow returning to the apparatus from the chamber.

The air flow, containing hydrogen peroxide, is preferably redirected from its normal path to the second sensor. The sensor serves two purposes: a) it enables the apparatus to determine when the required concentration of the hydrogen peroxide has been reached so that a "killing period" (sterilization period) can begin and b) it enables the apparatus to determine when the hydrogen peroxide concentration has been reduced to a safe level at the end of the cycle.

The means for removing the hydrogen peroxide from the air flow is preferably a catalyst such as a metal catalyst which converts hydrogen peroxide into water and oxygen. Examples of such catalysts include a platinum/alumina catalyst and a ruthenium/alumina catalyst.

The "killing period" is the period of time it takes a specified concentration of hydrogen peroxide vapor to kill all biological matter. This time is inversely proportional to the concentration required. providing the concentration is over 400ppm, the product of the time (in seconds) and the concentration (in ppm) is preferably at least 180,000.

It is preferred that the hydrogen peroxide concentration is maintained at at least 600ppm, more preferably at least 900ppm, in the chamber during the "killing period", although much higher concentrations may be employed. For safety reasons it is necessary, at the end of the cycle, to remove the hydrogen peroxide from the air flow.

It is therefore desirable to provide a sensor that can accurately and consistently measure high concentrations <u>and</u> low concentrations of hydrogen peroxide. However known hydrogen peroxide sensors are only able to make sensitive measurements within a narrow concentration range.

The present invention has overcome this problem by providing a dilution means for the means for monitoring the hydrogen peroxide concentration. The dilution

means causes a dilution gas, such as air, to join a sampled air flow from the air exiting the chamber before it reaches the sensor, thereby diluting the hydrogen peroxide concentration in a known ratio. The ratio of the dilution gas to the sampled air returning from the chamber can be determined by means of a pump or pumps and orifice plates which fix in each case the volume of air passing. The sensor accurately measures the concentration of the hydrogen peroxide, since it is maintained within its sensitivity range, and the results are calculated taking into account the dilution ratio. The whole monitoring system may be fully automated.

When the concentration of hydrogen peroxide has been reduced at the end of the cycle by the catalyst, the remaining concentration is measured with the dilution means inoperable, to determine that a safe level has been reached.

The present applicants have also found that more accurate measurements of the hydrogen peroxide concentration can be obtained if the pathway of the gas flow to the sensor is heated, as this prevents the hydrogen peroxide condensing out. It has also been found desirable to heat the pathway of the dilution air entering the system via the diluter, as the ratio of the external air to that being diluted can be in the region of 300:1.

The pathways are preferably pipes and may be heated by wires coiled around them.

In yet another aspect therefore the invention provides a device for sensing hydrogen peroxide concentration, having a sensor for sensing hydrogen peroxide concentration in a gas, an inlet pipe for passing a gas being measured to said sensor and heating means for heating said inlet pipe so as to heat gas therein. Preferably the device includes dilution means for injecting a diluent gas into said inlet pipe to dilute a gas being measured before it reaches said sensor, said dilution means including means for heating said diluent gas.

Embodiments of the invention will now be described by way of example, with reference to the accompanying drawings.

In the drawings:

Fig. 1 is a diagram of an apparatus according to the present invention.

Fig. 2 is a diagram of a device for sensing hydrogen peroxide concentration according to a another aspect of the present invention.

In Fig. 1 there is shown part of the wall 2 of a chamber 1 which is to be sterilized. This chamber may be for example part of a pharmaceutical production or packaging line. It is connected by detachable flexible hoses 3,42 to the sterilizing apparatus embodying the invention. In a sterilizing operation, air is circulated through the chamber 1 and the sterilizing apparatus in a continuous flow.

The outlet hose 3 is connected to an air filter 4 which removes contaminants such as bacteria and is for example a Microflow HEPA filter. Downstream of the filter 4 in the main air circulating flow is a catalyst chamber 6 containing a suitable metal catalyst for decomposing hydrogen peroxide in the air to water and oxygen.

The catalyst chamber 6 is connected to a blower fan 7 which drives the circulating air around the system. The fan 7 is connected to a dehumidification system having three air cooling heat exchanger refrigerator devices 8,13,14. The first device 8 connected to a coolant compressor 9 cools the air to about 10°C. From this device the air passes to a valve 12 which directs it alternately to the parallel-connected refrigerant devices 13 and 14 which reduce the temperature to below 0°C to remove moisture as ice. The devices 13 and 14 are connected to a coolant compressor 34 via valves 35,36 and are each provided with a defrosting heater 15,16 which operates when the device is switched out of line. Condensed water is removed from the three devices 8,13,14 by pumps 10 to a container 11.

Downstream of the dehumidification system is a first air re-heater 17 from which the dried air passes through an orifice plate 18 across which a pressure sensor 19 is connected for measurement of the circulating air flow rate, which is required for control of the rate of hydrogen peroxide generation.

For control of flow rate and admission of external air and venting of air there are provided an admission valve 22 which admits external air through an absolute filter 40 upstream of the fan 7 and a venting valve 20 downstream of the orifice plate 18 which vents air to atmosphere via a filter 21.

The air circulating path downstream of the orifice plate 18 has a second air reheater 23 which raises air temperature to 50°C and then the hydrogen peroxide gas generator 24 which is of a known type containing heated plates onto which is fed a solution of hydrogen peroxide in water. The water and hydrogen peroxide are thus evaporated into the warm air. The generator is fed from a storage bottle for the solution by three peristaltic pumps 25,26 and 27 which allow fine control of the solution flow rate. The gas generator 24 is connected by the detachable flexible hose 42 to the chamber 1.

A pressure transducer 37 is also connected detachably to the chamber 1 via a hose 45, and senses the pressure in the chamber 1.

Fig. 1 also shows a relative humidity sensor 29 of conventional type, e.g. humidity sensor from Vaisala, Helsinki, Finland, connected to the circulating air flow downstream of the filter 4 by a branch line containing a shut-off valve 43 for isolation of the sensor 29 when hydrogen peroxide is passing, to prevent damage to it.

A hydrogen peroxide concentration monitor 5 is also connected to the circulating air flow between the filter 4 and the catalyst 6 via input pipe 5a. This monitor 5 is capable of measuring accurately the hydrogen peroxide concentration in air over a range of 0 to 3 ppm, and is in this case the EGM Exhaust Gas Monitor made by MDA Scientific Inc., Illinois, USA. A fixed volume flow rate of sampled air from the circulating air flow is established by an orifice plate 33 and a downstream pump

33a. Downstream of the pump 33a, a dilution system 30 is connected to the input pipe 5a, in order to feed in dilution air taken from the atmosphere via an absolute filter 31. A fixed flow rate of dilution air is established by an orifice plate 32 and a pump 32a, so that a predetermined dilution ratio, e.g. of 300:1, is obtained when the pump 32a operates. If desired, the dilution system 30 can be designed to provide an adjustable dilution ratio.

The whole length of the input pipe 5a and the dilution air piping through the dilution system 30 are both provided with electrical resistance heaters in the form of heater wires embedded in insulating tapes wound around them, in order that they can be maintained at a desired temperature. Particularly, the temperature of the dilution air entering the input pipe 5a is preferably at least 50 to 60°C.

As shown, the air passing through the monitor 5 is vented to atmosphere through a hydrogen peroxide decomposition catalyst 44.

PTFE tubing is generally used for flowpaths where hydrogen peroxide passes, since this has been found to minimize decomposition of the hydrogen peroxide.

The sterilizing apparatus of Fig. 1 is monitored and controlled by a control device, incorporating a microprocessor, which for clarity is not shown in the Figure. The control device receives as inputs:

(1) the hydrogen peroxide concentration sensed by the monitor 5,
(2) the relative humidity sensed by the sensor 29,
(3) the chamber pressure sensed by the sensor 37,
(4) the pressure drop sensed by the sensor 19.

From these inputs, from stored data and programs and from operator input concerning the desired sterilization performance, the control device calculates and executes the required control of the apparatus, in particular:

(1) the operation of the pump 32a of the dilution system 30,
(2) the operation of the valve 43,
(3) the operation of the valves 20,22 to admit or vent air,
(4) the operation of the valve 12 and the valves 35,36 to switch between the cooling devices 13,14,
(5) the pumps 25,26,27 to control hydrogen peroxide generation,
(6) the fan 7.

The method of operation of the apparatus, under this automatic control by the control device, is described below.

The sterilization of a chamber by the method of the invention has four phases.

1. Conditioning:

During this phase the relative humidity in the chamber is reduced to bring it below a predetermined threshold, for example 10%. This is necessary to allow the full amount of the aqueous solution of hydrogen peroxide to be evaporated, which by the nature of the process increases the humidity. The returned air from the chamber is passed through the dehumidification system 8, 13, 14 within the apparatus which removes the water vapor.

2. Raising the hydrogen peroxide gas concentration:

After conditioning, the gas generator 24 is started to generate hydrogen peroxide gas which is passed into the chamber 1. As the gas/air mixture which is supplied by the generator 24 is mixed with the air in the chamber the gas concentration in the chamber rises. When the hydrogen peroxide gas concentration in the chamber has reached the desired value, e.g. 900 ppm, the effective sterilizing period begins.

3. Maintaining the hydrogen peroxide gas concentration:

The desired gas concentration in the chamber 1 is maintained for a predetermined time sufficient to achieve the required sterilization effect.

4. Aeration:

After the sterilization period the hydrogen peroxide gas is removed from the chamber. Typically, for health and safety reasons, it is necessary to reduce the concentration of hydrogen peroxide gas to a level of 1 ppm so that the chamber can be returned to its normal use.

These four phases are discussed in more detail below with reference to Fig 1.

1. Conditioning:

Air is pumped from the chamber 1 by the fan 7 via the flexible hose 3. The air passes through the filter 4, e.g. a Microflow HEPA filter, to protect internal components of the apparatus from contamination.

The fan 7 moves the air to the first stage refrigerator device 8 which cools it to approx 10°C. The second stage refrigerator devices 13, 14, operating alternately, reduce the temperature to below 0°C, to remove more water.

Air is then reheated by heater 17 to a moderate temperature in order that the air pressure and therefore flow rate can be accurately measured. This is important as it will gov rn the quantity of hydrogen peroxide that is released into the air stream during the second phase. The dry air enters the chamber 1.

The relative humidity of the air is accurately measured at any time during this stage by the sensor 29 which

tests the air returning from the chamber 1. When not required, ie during phases 2, 3 and 4, the sensor 29 is isolated by valve 43 to protect it from the hydrogen peroxide. When the required low level of selective humidity has been detected by the sensor 29, the control device of the apparatus starts phase 2.

2. Raising the hydrogen peroxide gas concentration.

The hydrogen peroxide concentration within the chamber is very important. As described above, a predetermined concentration is maintained for a sufficient period of time for the sterilization to take place. For example, microbiological data has been obtained based on gas concentrations of 900ppm and hence the "killing period" can be started when this concentration has been reached.

To measure the air flow rate the air is passed through the orifice plate 18. The pressure across the plate 18 is measured by the pressure transducer 19 and fed to the control device which calculates the flow rate. The airflow can be increased or decreased as necessary. The control device can via control units 38 and 39 allow air to be added or vented via valves 20, 22 and filters 21,40 respectively, and the control device adapts air flow rate by increasing or decreasing the speed of the fan 7. The pressure transducer 37 measures the pressure within the chamber 1 which typically is maintained at slightly above atmospheric by the control device of the apparatus.

The reheater 23 raises the air temperature to 50°C.

A 30% solution of hydrogen peroxide is stored in the bottle 28. A suitable source for this sterilant is BDH Merck Ltd. The gas generator 24 is fed by the three peristaltic pumps 25, 26 and 27. The use of three peristaltic pumps allows more sensitive control of the release of hydrogen peroxide into the air stream. The air/gas mixture then enters the chamber 1 via flexible hose 42.

The feed rate of the hydrogen peroxide solution to the generator 24 is determined by the control device of the apparatus in dependance of the air flow rate through the generator 24, in order to achieve a maximum rate of hydrogen peroxide generation.

The gas sensor 5 continuously measures the concentration of hydrogen peroxide returning from the chamber 1. The preferred sensor is manufactured by MDA Corporation, USA, and can measure accurately hydrogen peroxide concentrations of 0 ppm to 3 ppm. However the concentration in the chamber may be up to 1000 ppm. Therefore the diluter 30 adds ambient air to the gas stream passing to the sensor 5 at a known rate so that the concentration of hydrogen peroxide returning from the chamber 1 can be calculated.

A typical dilution ratio of dilution air/sampled gas is 300:1.

The pipe carrying the ambient dilution air and the pipe carrying the sampled mixture to the sensor 5 are heated by resistance h ating wires 41. This heating prevents absorption of hydrogen peroxide by the piping and condensation of the hydrogen peroxide and has been found to improve the accuracy of the concentration measurements.

The sensor 5 thus detects the rise of hydrogen peroxide concentration in the chamber 1. When the predetermined level is reached, the control device of the apparatus moves to phase 3.

3. Maintaining the gas concentration:

The required concentration of hydrogen peroxide is maintained for the necessary "killing period".

This is achieved by the control device of the apparatus by control of the generator 24 in dependence on the concentration sensed by the sensor 5.

4. Aeration:

At the end of the "killing period" the control device stops the gas generator 24, and a purge period commences, during which the air circulation through the apparatus continues. The catalyst 6 removes the hydrogen peroxide. Examples of suitable catalysts include platinum/alumina catalyst and a ruthenium /alumina catalyst.

The purge time, i.e. the time to clear the hydrogen peroxide to a desired level, will depend on the size of the chamber 1. The sensor 5 measures the concentration of the hydrogen peroxide first with operation of the diluter 30 until the sensed concentration falls below a suitable value such as 20 ppm, which does not damage the sensor 5. At this concentration, the diluter 30 is switched off by the control device and the sensor 5 will measure the concentration accurately from 3ppm down to as low as 0.1 ppm. When the control device has determined that the required low concentration level e.g. 1 ppm, has been reached, the chamber 1 is ready to be returned to its normal use. At this point, the control device may give a signal, such as an audible or visual signal, that sterilization is complete, and manually or automatically a normal air circulation means for the chamber 1 may be substituted for the apparatus of Fig. 1, which can then be disconnected.

Throughout all four phases, the catalyst 6 is in the air flow, and removes all hydrogen peroxide exiting from the chamber 1, to prevent damage to the downstream components. Likewise, the dehumidification system 8, 13, 14 and heaters 17, 23 are operating at all times, so that air at a desired temperature, free of hydrogen peroxide, is passed at a known flow rate to the generator 24.

Fig. 2 shows a diagram of a device for sensing hydrogen peroxide concentration. This device can be incorporated into the sterilising apparatus of Fig. 1 in substitution for the parts 4,5,6,30, 44 shown in Fig. 1.

Dilution air enters the device via a high efficiency filter 107. This filter 107 ensures that the device remains clean and substantially free of particulate matter and

therefore reduces the likelihood of hydrogen peroxide decay. An adjustable orifice 109 is adjusted and set during validation and calibration prior to use of the device so that the amount of flow can be determined and thus controlled.

A gas, e.g. air, which is being monitored and may for example contain hydrogen peroxide in a concentration anywhere between 0 and 2000 ppm enters the device via inlet 150. The main flow of this air passes through a hydrogen peroxide decomposition catalyst 104 and, now free of hydrogen peroxide gas, is directed back into the sterilizing apparatus via outlet 160. The catalyst 104 is protected from contamination by a high efficiency filter 151.

In order to measure the concentration of hydrogen peroxide in this air, a sample of the air containing hydrogen peroxide (sample air) passes through either a first orifice 106 or a second orifice 105 from the inlet side of the filter 151.

If the concentration of hydrogen peroxide is high and therefore dilution air is required, valve 110 is closed and valve 108 is open. The sample air therefore passes through the first orifice 106 and joins the stream of dilution air.

If the concentration of hydrogen peroxide is low and no dilution air is required, valve 108 is closed and valve 110 is open. The sample air then passes through the second orifice 105. The valves 108, 110 are under the control of control units 108a and 110a which are controlled by the control device of the sterilising apparatus.

The tubing 140 carrying the sample air and the dilution air is insulated and heated by a heater 111. Heating is important as the hydrogen peroxide gas otherwise readily creates a film on the internal surfaces of the tubing. It can be provide by resistance wire tape heater 111 surrounding the tubing 140. A suitable temperature of the tubing is 50 - 60°C.

Two diaphragm pumps 100 in parallel move the sample air, with or without the dilution air, through the device. The sample air is passed through a monitor 102 such as the EGM Exhaust Gas Monitor manufactured by MDA Scientific Inc or a Polytron manufactured by Drager. The sample air, having passed through the monitor 102 is then vented to atmosphere through a hydrogen peroxide decomposition catalyst 103. The hydrogen peroxide decomposition catalyst 103 is fitted with an inlet filter 130 to protect the catalyst and an outlet filter 170 to protect personnel in the environment from dust which may be shed from the catalyst.

In Fig. 2, there is shown diagrammatically a multiway valve 117, which under control of the control device of the sterilizing apparatus, directs the air flow from the pumps 100 to the sensor 102 or directly to the catalyst 103. The sensor 102 may thus be isolated at appropriate times. This valve 107 is also heated by a resistance wire tape heater 111.

Claims

1. A sterilizing apparatus for circulating gas through a chamber to be sterilized, said apparatus comprising

   means for circulating the gas
   at least one dehumidifier for the circulating gas
   means for introducing hydrogen peroxide into the gas flow prior to it entering the chamber, and
   means for removing hydrogen peroxide from the gas flow after it leaves the chamber;
   a first sensor for monitoring the relative humidity of the gas after it leaves the chamber;
   a second sensor for monitoring the concentration of hydrogen peroxide of the gas after it leaves the chamber; and
   control means arranged to control said means for introducing hydrogen peroxide in dependence on the outputs of said first and second sensors.

2. A sterilizing apparatus according to claim 1 wherein the first sensor for monitoring the relative humidity of the gas flow is positioned such that gas returning to the apparatus can be monitored.

3. A sterilizing apparatus according to any one of the preceding claims wherein only a sample of the gas returning from the chamber is directed to the first sensor to be monitored.

4. A sterilizing apparatus according to claim 3 wherein a valve controls when a sample is directed to the first sensor.

5. A sterilizing apparatus according to any one of the preceding claims wherein the second sensor for monitoring the hydrogen peroxide concentration is positioned to monitor the gas flow returning to the apparatus from the chamber.

6. A sterilizing apparatus according to any one of the preceding claims wherein the second sensor further comprises a dilution means for introducing a dilution gas into the gas flow from the chamber before it reaches the sensor.

7. A sterilizing apparatus according to claim 6 wherein the ratio of dilution gas to gas flow returning from the chamber is determined by a pump and orifice plates which fix the volume of gas passing therethrough.

8. A sterilizing apparatus according to claim 7 wherein the dilution means and the second sensor are fully automated.

9. A sterilizing apparatus according to any one of claims 6, 7 or 8 wherein the pathway of dilution gas from the diluting means to the sensor is heated.

10. A sterilizing apparatus according to any one of the preceding claims wherein the pathway of the gas flow from the chamber to the second sensor is heated.

11. A sterilizing apparatus according to any one of the preceding claims wherein the means for removing the hydrogen peroxide from the gas flow is a catalyst.

12. A sterilizing assembly having a chamber to be sterilised and an apparatus for circulating gas through the chamber, said apparatus comprising

means for circulating the gas
at least one dehumidifier for the circulating gas
means for introducing hydrogen peroxide into the gas flow prior to it entering the chamber, and
means for removing hydrogen peroxide from the gas flow after it leaves the chamber;
a first sensor for monitoring the relative humidity of the gas in the chamber;
a second sensor for monitoring the concentration of hydrogen peroxide of the gas in the chamber; and
control means adapted and arranged to control said means for introducing hydrogen peroxide in dependence on the outputs of said first and second sensors.

13. A sterilizing apparatus according to claim 12 wherein the first sensor for monitoring the relative humidity of the gas flow is positioned such that gas returning to the apparatus can be monitored.

14. A sterilizing apparatus according to claim 12 or claim 13 wherein only a sample of the gas returning from the chamber is directed to the first sensor to be monitored.

15. A sterilizing apparatus according to claim 14 wherein a valve controls when a sample is directed to the first sensor.

16. A sterilizing apparatus according to any one of claims 12 to 15 wherein the second sensor for monitoring the hydrogen peroxide concentration is positioned to monitor the gas flow returning to the apparatus from the chamber.

17. A sterilizing apparatus according to any one of claims 12 to 16 wherein the second sensor further comprises a dilution means for introducing a dilution gas into the gas flow from the chamber before it reaches the sensor.

18. A sterilizing apparatus according to claim 17 wherein the ratio of dilution gas to gas flow returning from the chamber is determined by a pump and orifice plates which fix the volume of gas passing therethrough.

19. A sterilizing apparatus according to claim 18 wherein the dilution means and the second sensor are fully automated.

20. A sterilizing apparatus according to any one of claims 17, 18 or 19 wherein the pathway of dilution gas from the diluting means to the sensor is heated.

21. A sterilizing apparatus according to any one of claims 12 to 20 wherein the pathway of the gas flow from the chamber to the second sensor is heated.

22. A method of sterilizing a chamber using hydrogen peroxide as sterilant, in which the gas of the atmosphere in the chamber is cycled through apparatus adapted to dehumidify it, add hydrogen peroxide gas to it and remove hydrogen peroxide gas from it, the method comprising the steps of

(1) dehumidifying the gas in said apparatus and sensing the humidity of the atmosphere in the chamber to detect when a predetermined low humidity value is obtained,
(2) when said predetermined humidity value is obtained, adding hydrogen peroxide gas to the gas in said apparatus and sensing the hydrogen peroxide concentration in the atmosphere in the chamber to detect when a predetermined first hydrogen peroxide concentration value is obtained,
(3) when said predetermined first hydrogen peroxide concentration value has been obtained, maintaining the hydrogen peroxide concentration in the atmosphere in the chamber at at least a further predetermined value, which may be the same as said first value, for a predetermined period of time, by sensing the hydrogen peroxide concentration in the atmosphere in the chamber and adding further hydrogen peroxide gas to the gas in said apparatus as required, and
(4) after said predetermined period of time, removing hydrogen peroxide from the gas in said apparatus and sensing the hydrogen peroxide concentration in the chamber to detect when a predetermined low value thereof is obtained.

23. A device for sensing hydrogen peroxide concentration, comprising a sensor for sensing hydrogen per-

oxide concentration in a gas, an inlet pipe for passing a gas being measured to said sensor, and heating means for heating said inlet pipe so as to heat gas therein.

24. A device according to claim 23 further comprising a dilution means for injecting a diluent gas into said inlet pipe to dilute a gas being measured before it reaches said sensor.

25. A device according to claim 24 wherein said dilution means includes means for heating said diluent gas.

26. A control system for controlling a sterilizing apparatus for circulating gas through a chamber to be sterilized by hydrogen peroxide comprising

an inlet pipe for receiving gas from the chamber;
a first sensor for monitoring the relative humidity of the gas after it leaves the chamber;
a second sensor for monitoring the concentration of hydrogen peroxide of the gas after it leaves the chamber; and
a control means adapted and arranged to generate a signal to control the sterilising apparatus, the signal being dependent on the outputs of said first and second sensors.

27. A method of controlling a sterilizing apparatus for circulating gas through a chamber to be sterilised comprising the steps of

circulating the gas through the chamber;
dehumidifying the circulating gas;
monitoring the relative humidity of the gas after it leaves the chamber by means of a first sensor;
introducing hydrogen peroxide into the gas flow prior to it entering the chamber;
removing hydrogen peroxide from the gas flow after it leaves the chamber;
monitoring the concentration of hydrogen peroxide of the gas after it leaves the chamber by means of a second sensor; and
controlling the introduction of hydrogen peroxide in dependence on the outputs of the first and the second sensors.

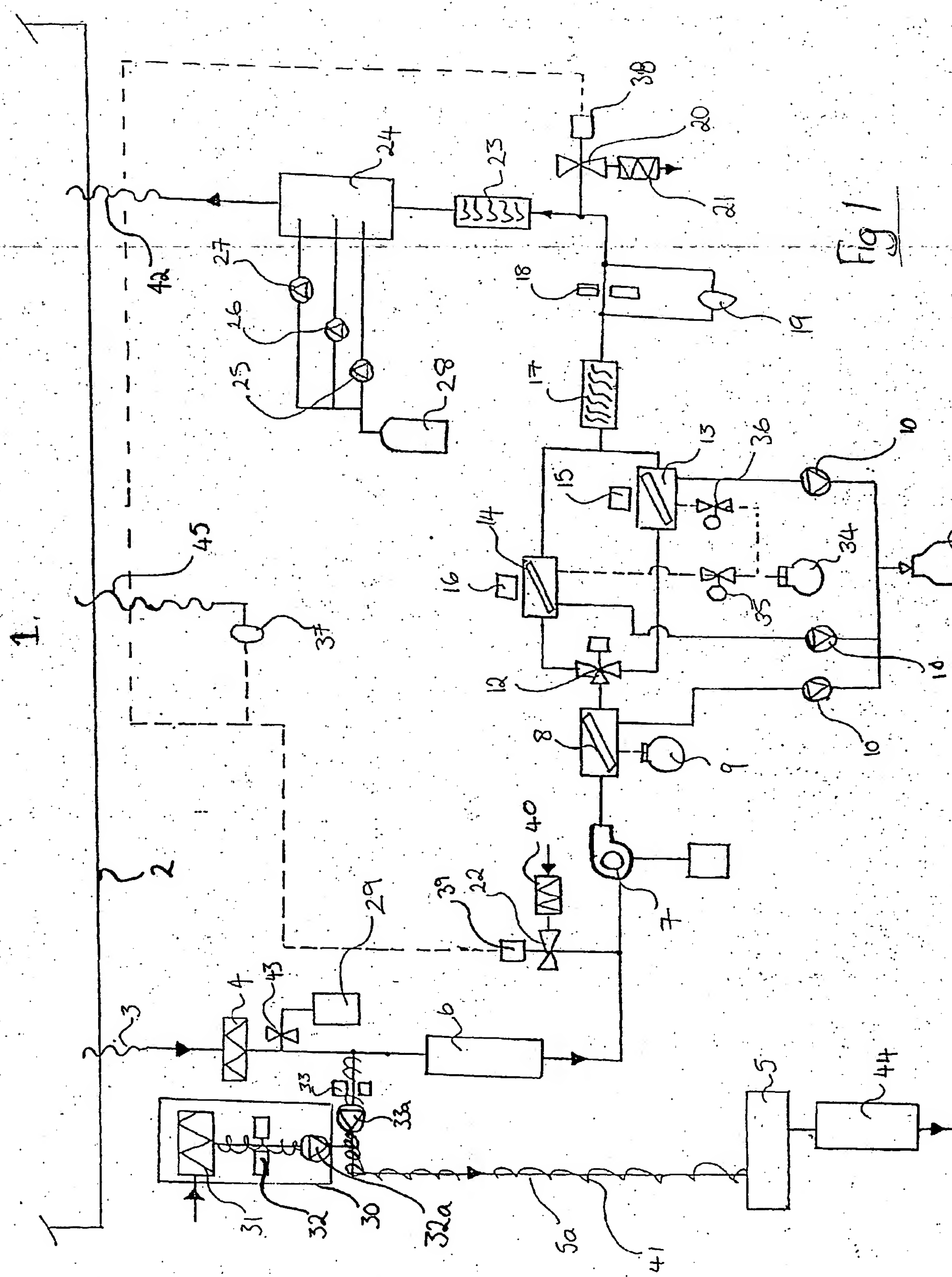
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Fig 1

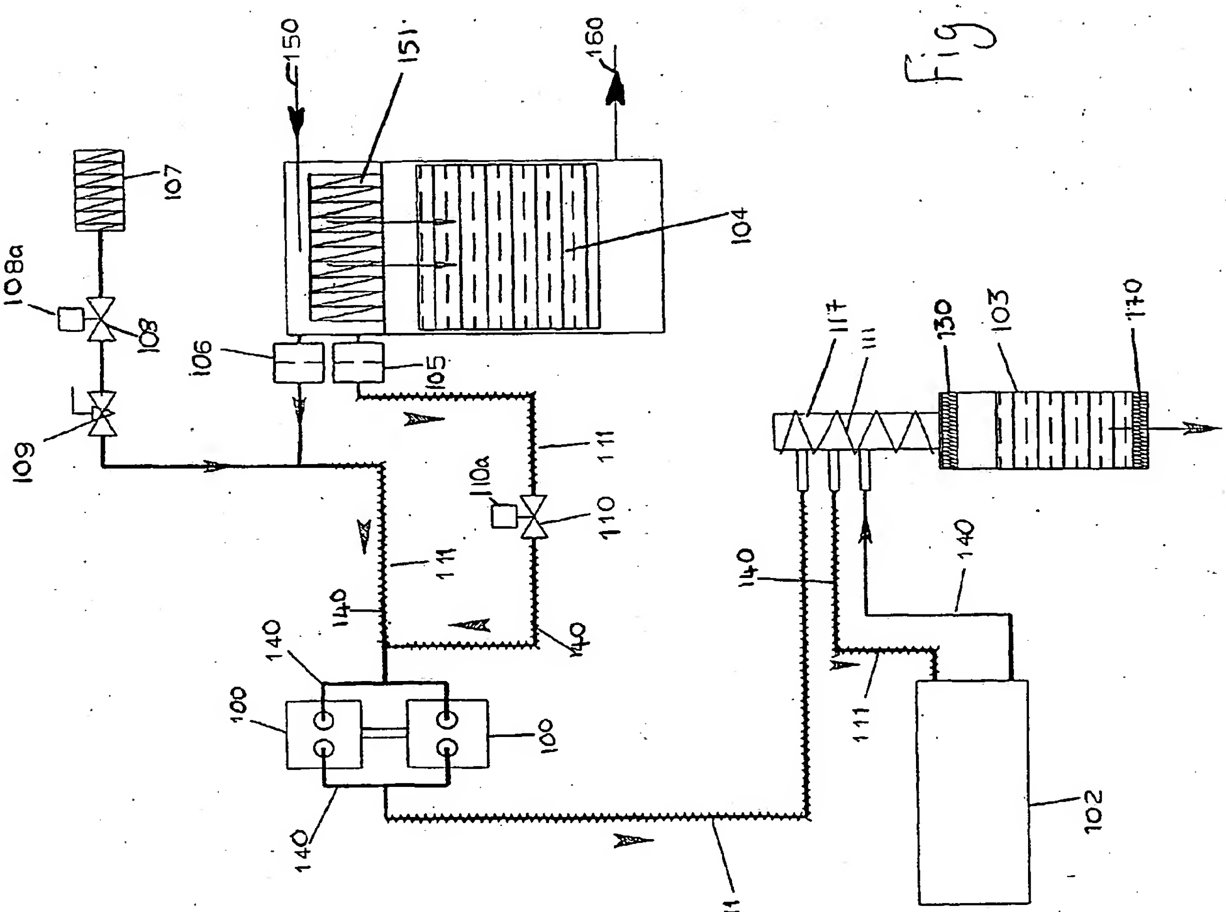
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Fig. 2

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 96 30 8398

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	WO 91 05573 A (AMERICAN STERILIZER) * claims 1-25 *	1	A61L2/20 A61L2/24
A	EP 0 452 780 A (ABBOTT) * claims 1-10 *	1	
A	EP 0 384 535 A (SHIKOKU KAKOKI) * claims 1-7 *	1	

TECHNICAL FIELDS SEARCHED (Int.Cl.6)

A61L

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	19 February 1997	Peltre, C

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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